1,050,497

PATENT SPECIFICATION

NO DRAWINGS

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Date of Application and filing Complete Specification: March 3, 1965.

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COMPLETE SPECIFICATION

Process for the Reaction of Fats free from Active Hydrogen Atoms with Alkylene Oxides

SPECIFICATION NO. 1,050,497

The inventors of this invention in the sense of being the actual devisers thereof within the meaning of Section 16 of the Patents Act, 1949 are: Gunther Boekmke, Roggendorfstrasse 85, Koln-Flittard, Germany, Mathieu Quaedvlieg, v.Bottingerstrasse Leverkusen, Germany, both of German nationality.

THE PATENT OFFICE

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oleate, and boron fluoride.

In general, it is advisable to maintain a temperature of about 100—180°C. during the reaction of the alkylene oxides with the fats.

Fats which are free from active hydrogen atoms, are, for example, the following: beef tallow, lard, coconut fat and waxes, as well as olive oil, cotton seed oil, peanut oil, palm kernel oil, soya bean oil, poppy seed oil, sunflower seed oil, fish oils, whale oil and bone oils; they also include the solid fats obtainable from liquid fats containing double bonds by means of hydrogenation.

The reaction products obtained according to the process of the present invention have excellent surface-active properties and may, therefore, be successfully used as dispersing agents, emulsifying agents, detergents, plasticisers, levelling agents and defoaming agents.

The proportions by weight of the alkylene oxides in relation to the fats may vary within wide limits during the process according to the present invention; the proportions by weight which are expedient in a given case for a specific application can readily be established by preliminary experiments. When, for example, the reaction products are intended for use as dispersing and emulsifying agents or as detergents, it is generally expedient to react 5—15 moles or 10—25 moles of ethylene oxide, respectively, per 1 equivalent of the fat acids contained in the fats; however, when it is intended to use the reaction products as defoaming agents, it is generally advisable to react 3—10 moles of propylene oxide per 1 equivalent of the fat acids contained in the fats.

It is known to react fats which are free from active hydrogen atoms, with ethylene oxide in the presence of sodium hydroxide as catalyst but the speed of the reaction is exceedingly slow. In contradistinction thereto, the speed of the reaction is considerably increased in the process of the present invention.

Furthermore, the reaction products obtained according to the present invention are distinguished by their more marked surface-active properties, compared with the products of the action of alkylene oxide based on fats having active hydrogen atoms, such as wool fat, castor oil and partially saponified glycerides of fat acids. This is mainly evidenced when the products are used as emulsifying agents and as detergents.

Furthermore, it is remarkable that pharmaceutical preparations which contain the

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COMPLETE SPECIFICATION

Process for the Reaction of Fats free from Active Hydrogen Atoms with Alkylene Oxides

We, FARBENFABRIKEN BAYER AKTIENGESELLSCHAFT, a body corporate organised under the laws of Germany, of Leverkusen, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention is concerned with a process for the reaction of fats which are free from active hydrogen atoms, with alkylene oxides.

Thus, according to the present invention, there is provided a process for the reaction of fats which are free from active hydrogen, with alkylene 1,2- oxides, such as ethylene oxide, propylene oxide and butylene oxide, in the presence of fat-soluble oxyalkylation catalysts.

The following may be mentioned as examples of fat soluble oxyalkylation catalysts: alkali metal and alkaline earth metal alkoxides, such as sodium butoxide, sodium dodecyloxide and sodium tetraethylene glycoxide, the alkali metal and alkaline earth metal salts of fatty acids, such as potassium laurate, potassium stearate and potassium oleate, and boron fluoride.

In general, it is advisable to maintain a temperature of about 100—180°C. during the reaction of the alkylene oxides with the fats.

Fats which are free from active hydrogen atoms, are, for example, the following: beef tallow, lard, coconut fat and waxes, as well as olive oil, cotton seed oil, peanut oil, palm kernel oil, soya bean oil, poppy seed oil, sunflower seed oil, fish oils, whale oil and bone oils; they also include the solid fats obtainable from liquid fats containing double bonds by means of hydrogenation.

The reaction products obtained according to the process of the present invention have excellent surface-active properties and may, therefore, be successfully used as dispersing agents, emulsifying agents, detergents, plasticisers, levelling agents and defoaming agents.

The proportions by weight of the alkylene oxides in relation to the fats may vary within wide limits during the process according to the present invention; the proportions by weight which are expedient in a given case for a specific application can readily be established by preliminary experiments. When, for example, the reaction products are intended for use as dispersing and emulsifying agents or as detergents, it is generally expedient to react 5—15 moles or 10—25 moles of ethylene oxide, respectively, per 1 equivalent of the fat acids contained in the fats; however, when it is intended to use the reaction products as defoaming agents, it is generally advisable to react 3—10 moles of propylene oxide per 1 equivalent of the fat acids contained in the fats.

It is known to react fats which are free from active hydrogen atoms, with ethylene oxide in the presence of sodium hydroxide as catalyst but the speed of the reaction is exceedingly slow. In contradistinction thereto, the speed of the reaction is considerably increased in the process of the present invention.

Furthermore, the reaction products obtained according to the present invention are distinguished by their more marked surface-active properties, compared with the products of the action of alkylene oxide based on fats having active hydrogen atoms, such as wool fat, castor oil and partially saponified glycerides of fat acids. This is mainly evidenced when the products are used as emulsifying agents and as detergents.

Furthermore, it is remarkable that pharmaceutical preparations which contain the

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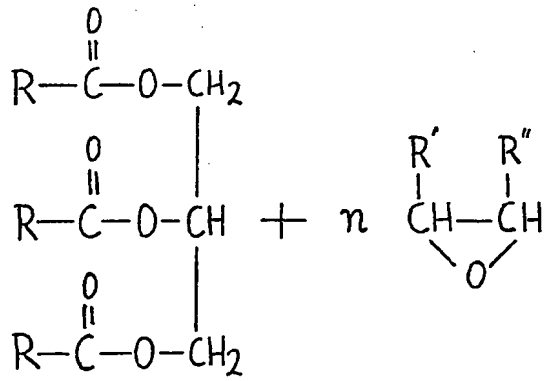
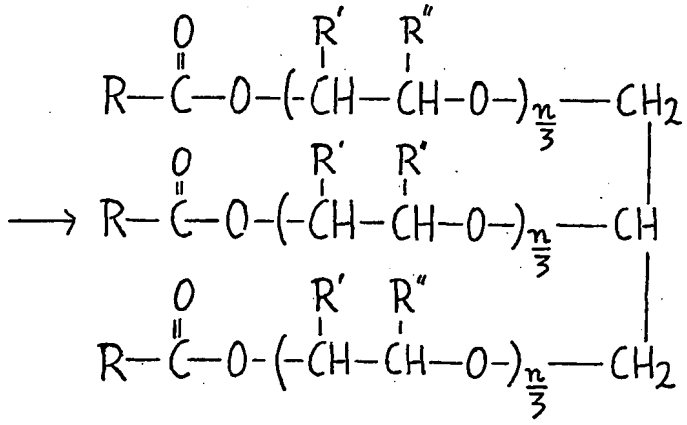
alkylene oxide reaction products obtained by the process according to the present invention are far better tolerated than preparations which contain alkylene oxide reaction products obtained by the known processes.

The action of the alkylene 1,2-oxides on fats free from active hydrogen atoms presumably proceeds, during the process according to the present invention, in accordance with the following equation:—





wherein R denotes a higher aliphatic hydrocarbon radical, R' and R'', which may be the same or different, stand for hydrogen atoms and/or methyl radicals and *n* represents a whole number of at least 3.

This hypothesis is based on the fact that the products prepared according to the process of the present invention only exhibit a very low OH-index. The fact that these products have an OH-index at all is likely to be due to the formation of a small amount of the reaction products of the alkylene oxides with fatty acids or with partially saponified fats as by-products.

The following Examples are given for the purpose of illustrating the present invention:—

EXAMPLE 1:

200 g. olive oil are mixed with 12 g. of a fat-soluble catalyst, which has been prepared from 10 g. oleic acid monoglyceride and 2 g. potassium hydroxide, and the mixture is heated *in vacuo* at 100—120°C. for 30 minutes in order to remove traces of moisture. Ethylene oxide is then passed into the mixture at 140—160°C. at atmospheric pressure until 300 g. have been taken up, this takes about 5 hours.

The reaction product thus obtained is soluble in oil and is exceedingly suitable for application as an emulsifying agent for vegetable or animal oils and for mineral oils. The product forms emulsions with water. When a 5% aqueous emulsion of the reaction

product is neutralised with acetic acid, two layers are obtained which have almost the same volume; the product is therefore capable of forming coazervates.

When 2.5 g. powdered potassium hydroxide are used in place of the fat-soluble catalyst prepared with the use of 2 g. potassium hydroxide, no absorption of ethylene oxide can be observed, even after heating at 140—160°C. has been continued for 9 hours.

EXAMPLE 2:

200 g. olive oil, which have been heated *in vacuo* at 100—120°C. for 30 minutes in order to remove traces of moisture, are mixed with 20 g. of a fat-soluble catalyst, which has been prepared from 18.5 g. tetraethylene glycol and 1.5 g. potassium and then reacted with 480 g. ethylene oxide as described in Example 1. The resultant reaction product, which is readily soluble in water and a 1% aqueous solution of which becomes turbid at 48°C., can be successfully used as an emulsifying agent or as a dispersing agent for pharmaceutical products.

EXAMPLE 3:

200 g. beef tallow (iodine number 38, acid number <1), which has been heated *in vacuo* at 100—120°C. for 30 minutes in order to remove traces of moisture, are melted and mixed with 20 g, of the fat-soluble catalyst described in Example 2. The mixture is then reacted with 85 g. ethylene oxide by the method described in Example 1. The resultant reaction product is soluble in oil and forms an emulsion with water. It can be used as a plasticiser for textile materials and as a defoaming agent in the sugar and paper industries.

EXAMPLE 4:

200 g. beef tallow (iodine number 38, acid number <1), which has been heated *in vacuo* at 100—120°C. for 30 minutes in order to remove traces of moisture, are heated at 160°C. with 10 g. fat-soluble potassium oleate. 300 g. ethylene oxide are then gradually caused to react with it at a pressure of 2 atmospheres. The product thus obtained, a 1% aqueous solution of which becomes turbid at 32°C. after it has been neutralised with acetic acid and a 5% aqueous solution of which shows signs of streak formation, has an excellent detergent activity, whilst forming very little foam.

EXAMPLE 5:

200 g. beef tallow (iodine number 38, acid number <1), which has been heated *in vacuo* at 100—120°C. for 30 minutes in order to remove traces of moisture, are melted with 20 g. of the fat-soluble catalyst described in Example 2 and the melt reacted with 420 g. ethylene oxide as described in Example 1. The reaction product, which is a solid and a 1% aqueous solution of which becomes turbid at 60°C., is an excellent levelling agent and detergent.

EXAMPLE 6:

300 g. beef tallow (iodine number 38, acid number <1), which has been heated *in vacuo* at 100—120°C. for 30 minutes in order to remove traces of moisture, are heated with 30 g. of a fat-soluble catalyst, prepared from 28 g. propylene glycol and 2 g. potassium, and with 174 g. propylene oxide in an autoclave, first of all at 100°C. for 2 hours and thereafter at 130°C. for 2 hours. A pressure increase of 5—6 atmospheres is experienced at the start and this pressure increase drops to 0 atmospheres at the end. The resultant oily reaction product can be emulsified in water and has a very good activity as a defoaming agent.

EXAMPLE 7:

200 g. coconut fat, which has been heated *in vacuo* at 100—120°C. for 30 minutes in order to remove traces of moisture, are mixed with 20 g. of the fat-soluble catalyst described in Example 2 and caused to react with 180 g. ethylene oxide as described in Example 1. The resultant product has a very good activity as a defoaming agent and as an emulsifier.

EXAMPLE 8:

200 g. coconut fat, which has been heated *in vacuo* at 100—120°C. for 30 minutes in order to remove traces of moisture, are mixed with 20 g. of the fat-soluble catalyst described in Example 2 and reacted with 500 g. ethylene oxide as described in Example 1. A very good detergent, with good foaming and wetting properties, is thus obtained.

WHAT WE CLAIM IS:—

1. The reaction products of fats which are free from active hydrogen atoms, with alkylene 1,2-oxides in the presence of fat soluble oxyalkylation catalysts.

2. Reaction products according to claim 1, which are hereinbefore specifically exemplified.

3. Process for the production of products according to Claim 1, wherein a fat which is free from active hydrogen atoms is reacted with an alkylnee 1,2-oxide in the presence of a fat-soluble oxyalkylation catalyst.

4. Process according to claim 1, wherein the reaction is carried out at a temperature of 100—180°C.

5. Process for the production of products according to Claim 1, substantially as hereinbefore described and exemplified.

6. Products according to claim 1, whenever prepared by the process according to any of claims 3 to 5.

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